

must be used to compute the total sulfur emission rate (E).

(c) Where compliance is achieved through the use of a reduction control system not followed by a continually operated incineration device, you must install, calibrate, maintain, and operate a continuous monitoring system to measure the emission rate of reduced sulfur compounds as SO<sub>2</sub> equivalent in the gases discharged to the atmosphere. The SO<sub>2</sub> equivalent compound emission rate must be expressed in terms of equivalent sulfur mass flow rates (kg/hr (lb/hr)). The span of this monitoring system must be set so that the equivalent emission limit of § 60.5405(b) will be between 30 and 70 percent of the measurement range of the system. This requirement becomes effective upon promulgation of a performance specification for continuous monitoring systems for total reduced sulfur compounds at sulfur recovery plants.

(d) For those sources required to comply with paragraph (b) or (c) of this section, you must calculate the average sulfur emission reduction efficiency achieved (R) for each 24-hour clock interval. The 24-hour interval

may begin and end at any selected clock time, but must be consistent. You must compute the 24-hour average reduction efficiency (R) based on the 24-hour average sulfur production rate (S) and sulfur emission rate (E), using the equation in § 60.5406(c)(1).

(1) You must use data obtained from the sulfur production rate monitoring device specified in paragraph (a) of this section to determine S.

(2) You must use data obtained from the sulfur emission rate monitoring systems specified in paragraphs (b) or (c) of this section to calculate a 24-hour average for the sulfur emission rate (E). The monitoring system must provide at least one data point in each successive 15-minute interval. You must use at least two data points to calculate each 1-hour average. You must use a minimum of 18 1-hour averages to compute each 24-hour average.

(e) In lieu of complying with paragraphs (b) or (c) of this section, those sources with a design capacity of less than 152 Mg/D (150 LT/D) of H<sub>2</sub>S expressed as sulfur may calculate the sulfur emission reduction efficiency achieved for each 24-hour period by:

$$R = \frac{K_2 S}{X}$$

Where:

R = The sulfur dioxide removal efficiency achieved during the 24-hour period, percent.

K<sub>2</sub> = Conversion factor, 0.02400 Mg/D per kg/hr (0.01071 LT/D per lb/hr).

S = The sulfur production rate during the 24-hour period, kg/hr (lb/hr).

X = The sulfur feed rate in the acid gas, Mg/D (LT/D).

(f) The monitoring devices required in paragraphs (b)(1), (b)(3) and (c) of this section must be calibrated at least annually according to the manufacturer's specifications, as required by § 60.13(b).

(g) The continuous emission monitoring systems required in paragraphs (b)(1), (b)(3), and (c) of this section must be subject to the emission monitoring requirements of § 60.13 of the

General Provisions. For conducting the continuous emission monitoring system performance evaluation required by § 60.13(c), Performance Specification 2 of appendix B to part 60 of this chapter must apply, and Method 6 must be used for systems required by paragraph (b) of this section.

**§ 60.5408 What is an optional procedure for measuring hydrogen sulfide in acid gas—Tutwiler Procedure?**

The Tutwiler procedure may be found in the Gas Engineers Handbook, Fuel Gas Engineering practices, The Industrial Press, 93 Worth Street, New York, NY, 1966, First Edition, Second Printing, page 6/25 (Docket A-80-20-A, Entry II-I-67).

(a) When an instantaneous sample is desired and  $\text{H}_2\text{S}$  concentration is ten grains per 1000 cubic foot or more, a 100 ml Tutwiler burette is used. For concentrations less than ten grains, a 500 ml Tutwiler burette and more dilute solutions are used. In principle, this method consists of titrating hydrogen sulfide in a gas sample directly with a standard solution of iodine.

(b) *Apparatus.* (See Figure 1 of this subpart) A 100 or 500 ml capacity Tutwiler burette, with two-way glass stopcock at bottom and three-way stopcock at top which connect either with inlet tubulature or glass-stoppered cylinder, 10 ml capacity, graduated in 0.1 ml subdivision; rubber tubing connecting burette with leveling bottle.

(c) *Reagents.* (1) Iodine stock solution, 0.1N. Weight 12.7 g iodine, and 20 to 25 g cp potassium iodide for each liter of solution. Dissolve KI in as little water as necessary; dissolve iodine in concentrated KI solution, make up to proper volume, and store in glass-stoppered brown glass bottle.

(2) Standard iodine solution, 1 ml=0.001771 g I. Transfer 33.7 ml of above 0.1N stock solution into a 250 ml volumetric flask; add water to mark and mix well. Then, for 100 ml sample of gas, 1 ml of standard iodine solution is equivalent to 100 grains  $\text{H}_2\text{S}$  per cubic foot of gas.

(3) Starch solution. Rub into a thin paste about one teaspoonful of wheat starch with a little water; pour into about a pint of boiling water; stir; let cool and decant off clear solution. Make fresh solution every few days.

(d) *Procedure.* Fill leveling bulb with starch solution. Raise (L), open cock (G), open (F) to (A), and close (F) when

solutions starts to run out of gas inlet. Close (G). Purge gas sampling line and connect with (A). Lower (L) and open (F) and (G). When liquid level is several ml past the 100 ml mark, close (G) and (F), and disconnect sampling tube. Open (G) and bring starch solution to 100 ml mark by raising (L); then close (G). Open (F) momentarily, to bring gas in burette to atmospheric pressure, and close (F). Open (G), bring liquid level down to 10 ml mark by lowering (L). Close (G), clamp rubber tubing near (E) and disconnect it from burette. Rinse graduated cylinder with a standard iodine solution (0.00171 g I per ml); fill cylinder and record reading. Introduce successive small amounts of iodine thru (F); shake well after each addition; continue until a faint permanent blue color is obtained. Record reading; subtract from previous reading, and call difference D.

(e) With every fresh stock of starch solution perform a blank test as follows: Introduce fresh starch solution into burette up to 100 ml mark. Close (F) and (G). Lower (L) and open (G). When liquid level reaches the 10 ml mark, close (G). With air in burette, titrate as during a test and up to same end point. Call ml of iodine used C. Then, Grains  $\text{H}_2\text{S}$  per 100 cubic foot of gas =  $100(D-C)$

(f) Greater sensitivity can be attained if a 500 ml capacity Tutwiler burette is used with a more dilute (0.001N) iodine solution. Concentrations less than 1.0 grains per 100 cubic foot can be determined in this way. Usually, the starch-iodine end point is much less distinct, and a blank determination of end point, with  $\text{H}_2\text{S}$ -free gas or air, is required.

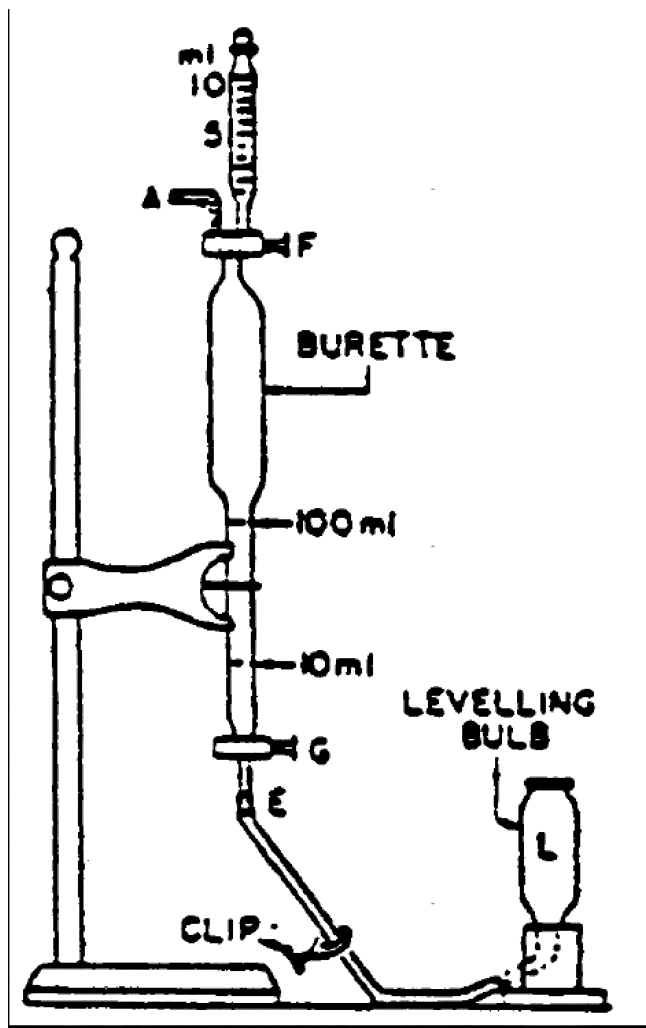


Figure 1. Tutwiler burette (lettered items mentioned in text).